

AMENDMENT NO. 3 JULY 2016

TO

IS 13965 : 1994 COCONUT MONOETHANOLAMIDE FOR COSMETIC INDUSTRY — SPECIFICATION

(Page 1, clause 2) — Insert the following references at the appropriate place:

<i>IS No.</i>	<i>Title</i>
695 : 1986	Specification for acetic acid (<i>third revision</i>)
1235 : 1988	Specification for acetic anhydride (<i>first revision</i>)
6804 : 1972	Specification for glass electrodes for direct reading pH meter

[Page 2, Table 1, *Sl No.* (xi)] — Insert the following new requirement:

(1)	(2)	(3)	(4)
xii)	Free monoethanolamine, percent by mass, <i>Max</i>	1.0	A-12

(Page 6, clause A-11.1.2) — Insert the following new clause at the end:

‘A-12 DETERMINATION OF FREE MONOETHANOLAMINE

A-12.0 Outline of the Method

Firstly, total free alkali (comprising of monoethanolamine, alkali and soap) is titrated with perchloric acid. Thereafter, monoethanolamine is blocked by acetylation of amino group with acetic anhydride. After blocking amino group, alkali and soap are titrated with perchloric acid. The free monoethanolamine is calculated using the formula.

A-12.1 Reagents

A-12.1.1 *Acetic Acid*, analytical reagent grade (see IS 695).

A-12.1.2 *Acetic Anhydride* — See IS 1235.

A-12.1.3 *Perchloric Acid Solution (0.1 N)* — Dissolve 8.5 ml of perchloric acid in 100 ml of acetic acid 1 litre amber glass bottle. Add 800 ml of acetic

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acid followed by 30 ml of acetic anhydride. Stopper the bottle and shake well. Standardize with potassium hydrogen phthalate potentiometrically.

A-12.2 Apparatus

A-12.2.1 *pH Meter*, provided with glass and calomel electrodes (see IS 6804).

A-12.3 Procedure

Weigh 6 g of material in a beaker to an accuracy of ± 0.0001 g and dissolve in acetic acid. Make the volume exactly to 100 ml in volumetric flask (Solution A). Pipette out 25 ml of solution A in a 250 ml beaker, add 50 ml of acetic acid. Titrate it potentiometrically with 0.1 N perchloric acid. Consider the burette reading as T_1 . Pipette out 25 ml of solution A in another 250 ml beaker. Add 10 ml of acetic anhydride and add 50 ml acetic acid. Close the beaker with aluminium foil and stir for 2 h. Titrate this solution with potentiometrically 0.1 N perchloric acid. Note the end point and consider this reading as T_2 .

A-12.4 Calculation

$$\text{Percent of free monoethanolamine} = \frac{[T_1 - T_2] \times N \times 4 \times 61}{10 \times M_1}$$

where

N = normality of perchloric acid;

T_1 = volume of 0.1 N perchloric acid used, in ml (without treatment of acetic anhydride);

T_2 = volume of 0.1 N perchloric acid used, in ml (with treatment of acetic anhydride); and

M_1 = mass of the material taken for the test. In g'.